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AFML-TR-71-250

**TENSILE, FRACTURE TOUGHNESS, AND  
CRACK GROWTH PROPERTIES OF  
A ROLL-EXTRUDED HP 9Ni-4Co-25C STEEL ALLOY**

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AIR FORCE MATERIALS LABORATORY  
AIR FORCE SYSTEMS COMMAND  
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

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## FOREWORD

This report was prepared by the University of Dayton Research Institute, Dayton, Ohio. The work was performed under USAF Contract No. F33615-71-C-1054. The contract was initiated under Project No. 7381, "Materials Applications," Task No. 738106, "Design Information Development," and administered by the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio, Mr. David C. Watson (AFML/LAE), Project Engineer.

All (or many) of the items compared in this report were commercial items that were not developed or manufactured to meet Government specifications, to withstand the tests to which they were subjected, or to operate as applied during this study. Any failure to meet the objectives of this study is no reflection on any of the commercial items discussed herein or on any manufacturer.

The author would like to acknowledge that testing performed for this program was accomplished by Messrs. R. J. Marton and J. H. Eblin. Engineering support was provided by Mr. G. J. Petrak. Test material was made available by Mr. P. Propp, A. F. Space and Missile System Office.

This report covers work conducted from April 1970 to April 1971. The contractor's report number is UDRI-TR-71-21.

This report was submitted by the author in May 1971.

This technical report has been reviewed and is approved.



A. OLEVITCH

Chief, Materials Engineering Branch  
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## ABSTRACT

A section of internal roll-extruded HP 9 Ni-4Co-25C steel alloy was obtained from an experimental 120-inch rocket motor case for Titan III and sent to the AFML for evaluation by the Space And Missile System Organization (SAMSO). Tensile and fracture toughness properties were obtained at room temperature and at -65°F. Crack growth properties were obtained at room temperature in laboratory air and in distilled water. An increase in tensile strength and a decrease in ductility were observed to occur with increased cold forming reduction. Fracture toughness properties appeared to decrease with increased rolling reduction. Fatigue crack growth properties in laboratory air were compatible with other high strength steels for the lower  $\Delta K$  values. Fatigue crack growth properties in distilled water showed no acceleration due to aqueous environment influences. Statically loaded stress corrosion crack (SCC) growth properties were an improvement over available D6ac steel alloy data.

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## SECTION I

### INTRODUCTION

A cold forming internal roll-extrusion process has been developed by the NTW (National Tapered Wing) Missile Engineering Company for the Manufacturing Technology Division of AFML under Air Force contract. The purpose of this development was to improve the fabrication of large diameter solid rocket motor (SRM) case segments for the Titan III Missile. Many alloys had been fabricated into SRM cases with varying degrees of success. Two of the materials suitable for this fabricating process, HP 9-4-25 and D6ac steel alloys, were selected for further processing examination. The results of the processing investigation by NTW indicated that the HP 9-4-25 alloy had higher potential because of its improved mechanical properties and ease of fabrication.

A section of the as-worked HP 9-4-25 steel was procured through SAMSO by the Materials Support Division of AFML for additional mechanical property evaluation by the University of Dayton Research Institute. It was the intent of this program to evaluate the effect of varying degrees of rolling reduction on the tensile and fracture toughness properties of HP 9-4-25 steel. Crack growth properties were also studied in some detail.

## SECTION II

### MATERIAL AND SPECIMENS

A 25 x 31-inch section was removed from a 120-inch diameter HP 9-4-25 test ring (S/N 004). The thinnest section of the ring had been reduced a total of 74% to a 0.423-inch thick section in five passes. One part of the ring was of varying thickness tapering from a 1.637-inch thick section to the highly worked 0.423-inch thick section (see Figure 1). The basic material was produced by Republic Steel with a vacuum arc remelt carbon-deoxidized process to meet the requirements of AMS 6541 material specification. The material was obtained from heat No. 3961419, and had the chemical composition shown in Table I.

The heat treatment of the forged preformed ring before and during internal roll-extrusion processing is presented in Table II.

All specimens were removed from the as-received material. In the terminology of this report, the "axial direction" is the long direction of the SRM case with longitudinal grain orientation and the "hoop direction" is the circumferential direction of the SRM case with transverse grain orientation. A photomicrograph of the grain structure in the highly worked 0.423-inch thick section of the SRM case is shown in Figure 2.

Tensile and fracture toughness properties were obtained using the specimen configurations shown in Figure 3 and Figure 4, respectively. By removing tensile specimens from the area of varying thickness in the axial and hoop directions it was possible to evaluate the effects of varying thickness on the resultant tensile properties. Compact tension fracture toughness specimens were machined from the axial (WR), hoop (RW), short-axial (WT), and short-hoop (RT) directions. WR and RW specimens were removed from the area of varying section thicknesses. The W, R, and T designations for fracture toughness specimens are in accordance with ASTM (see Figure 1).

Crack growth properties were studied using the Mostovsky<sup>1</sup> type (see Figure 5) tapered double cantilever beam (DCB) specimens and the elongated rectangular DCB specimens (see Figure 6). Two configurations of side grooves (semi-circular and V-shaped) were employed with the Mostovsky specimens. A side groove was not necessary with the rectangular DCB. Crack growth specimens were machined from the 0.4-inch-thick section in such orientation that the through-the-thickness cracks were propagated in the axial direction of the SRM case.

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<sup>1</sup>Mostovsky, et al., "Use of Crack-Line-Loaded Specimens for Measuring Plane-Strain Fracture Toughness," Journal of Materials, Vol. 2, No. 3, 1967.

TABLE I  
CHEMICAL ANALYSIS OF HP 9-4-25 STEEL ALLOY<sup>2</sup>

Chemical Composition (weight %)									
C	Mn	P	S	Si	Ni	Cr	Mo	Na	Co
0.28	0.29	0.008	0.009	0.02	8.40	0.44	0.50	0.09	3.95

TABLE II  
HEAT TREATMENT<sup>2</sup>

The heat treatment of rough forged ring (S/N 004) before internal roll-extrusion processing was as follows:

- A. Austenitized for 1 hour at 1650°F.
- B. Temperature dropped to 1,550 ±25°F for 2 hours.
- C. Oil quenched in room temperature oil.
- D. Furnace tempered at 1,000 ±25°F for 4 hours.
- E. Air-cooled.

After pass No. 3, an intermediate stress-relief cycle was utilized to reduce the internal hardness of the roll-extruded cylinder from  $R_C$  of 49 to an  $R_C$  of 44. The final configuration of the SRM segment was not heat treated.

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<sup>2</sup>Gott, J. E. and Lynch, J. M., "A Production Process for Large Solid Motor Cases Internal Roll-Extruded," AFML-TR-68-116, May, 1968.

### SECTION III

#### TEST PROCEDURES

Tensile and fracture toughness tests were performed with a Wiedemann tensile testing machine according to ASTM Standards at room and -65°F temperatures. Testing at -65°F was accomplished in a Conrad-Missimer environmental chamber using liquid nitrogen as a cooling agent.

Constant amplitude fatigue crack growth studies were performed using a closed-loop MTS hydraulic testing system. All cyclic loading wave forms were sinusoidal. Fatigue crack growth was monitored optically from the specimen surface with a 30X Gaertner traveling microscope. After specimen failure, the observed crack length was corrected for the internal curvature of the crack front. Both laboratory air and distilled water environments were used. A Plexiglas tank was used to contain the distilled water. The Mostovoy DCB specimen (in which the stress intensity factor,  $K_I$ , is essentially independent of crack length over a considerable range) and the rectangular DCB specimen (in which  $K_I$  is dependent upon the crack length) were utilized for these studies.

Statically loaded stress corrosion crack growth tests were performed on a Satec creep frame modified so as to load the specimen in the horizontal direction. This testing was accomplished in a Plexiglas tank containing distilled water. Crack opening displacement was monitored with a clip-on gage and converted to crack length using compliance measurements obtained from fatigue crack growth specimens. Again Mostovoy DCB and rectangular DCB specimens were tested. In all but one test, the specimens were precracked in the MTS system. The exception was a specimen with a corroded precrack which had not failed in a previous stress corrosion cracking (SCC) test. This specimen was statically loaded a second time until failure occurred.

## SECTION IV

### RESULTS AND DISCUSSION

The tensile properties developed for the HP 9-4-25 steel alloy are presented in Table III. In both the hoop and axial directions, the decrease in ductility and increase in strength at both room temperature and -65°F can be attributed to the cold working reduction process. At room temperature the ultimate strength was increased 22% in the hoop direction and 30% in the axial direction by the increased cold working required to reduce the section thickness from 1.5 inches to 0.4 inches. As can be observed, no highly directional tensile properties were detected.

Representative tensile specimens are shown in Figures 7, 8, and 9. These figures illustrate the laminations observed after testing the axial and hoop specimens removed from the 0.4-inch-thick heavily worked section. A photomicrograph of these laminations is shown in Figure 10. The distance between laminations in this figure was approximately 0.0004 inches.

The discontinuous laminar structure observed in fractured specimens is of considerable importance. It may be speculated that fracture toughness and crack growth properties through the radial wall thickness of the SRM case are improved due to the possibility of crack branching at the laminations. Further intermediate heat treatment during forming would be required to achieve a homogeneous material if desired.

The results of the fracture toughness tests are presented in Table IV. It can be seen in this table that valid  $K_{IC}$  values were not obtained from specimens removed from the 0.4-inch-thick section of the roll extrusion. This was because of insufficient material thickness. It can also be observed, however, that the toughness values calculated for the 0.4-inch-thick section (even though invalid) are sufficiently lower than those for the thicker section to say that the toughness is lower in the highly-worked thin section than in the thick section.

Both tensile and fracture toughness data were developed by Gott, et al<sup>2</sup>, for a plate which was removed from the material used to produce the forged preformed test ring. This plate had the same heat treatment as that given the rough forged preformed test ring prior to roll-extrusion processing. The fracture toughness values obtained by Gott, et al, from 1.88-inch-thick unrolled plate compared favorably with data obtained from the 1.4-inch-thick section in this report, although the 0.2% offset yield strength in the 1.4-inch-thick section was as much as 11% lower than the plate material before preforming.

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<sup>2</sup>Gott, J. E. and Lynch, J. M., "A Production Process for Large Solid Motor Cases Internal Roll-Extruded," AFML-TR-68-116, May 1968.

TABLE III  
TENSILE PROPERTIES OF HP 9-4-25 STEEL ALLOY  
SOLID ROCKET MOTOR CASE

Temperature (°F)	Direction (A)	Section Thickness (inches)	Ultimate Strength (KSI)	Yield Strength 0.2% Offset	Elongation in 1 inch G. L. (%)	Reduction Of Area (%)
Room	Hoop	1.5	170.1	156.8	15.7	50.2
		(B)	167.7	159.4	17.4	57.8
		(B)	172.1	168.0	16.7	57.2
		(B)	173.8	172.8	16.8	56.9
		0.8	186.6	173.4	11.9	50.7
		0.4	208.0	180.3	9.3	40.6
		0.4	210.6	188.1	9.2	42.5
		0.4	207.4	181.7	9.0	38.6
-65	Hoop	1.5	183.3	-----	16.2	48.8
		(B)	179.0	165.6	17.3	51.0
		(B)	174.9	164.1	18.2	53.9
		1.2	173.2	165.5	17.6	54.0
		0.4	210.5	178.6	9.1	35.5
		0.4	207.6	184.0	9.1	35.2
		0.4	208.3	178.9	9.8	36.2
Room	Axial	1.4(C)	166.7	158.4	16.4	54.9
		1.4(C)	166.7	159.6	16.4	53.3
		1.4(C)	167.4	158.1	16.6	55.0
		1.4(C)	167.7	159.4	17.1	54.7
		1.4(C)	167.4	159.1	16.4	55.0
		1.0(C)	177.0	177.0	20.0	59.7
		0.4	217.7	211.0	10.1	52.4
		0.4	217.4	209.6	9.7	49.7
		0.4	217.0	207.9	10.3	51.1
-65	Axial	1.4(C)	171.5	162.2	16.5	50.2
		1.0(C)	187.2	183.2	19.6	57.7
		1.0(C)	187.1	181.5	19.8	57.9
		0.4	214.7	210.6	11.0	49.7
		0.4	228.0	219.8	10.4	45.0
		0.4	227.9	223.8	10.4	43.5

(A) See explanation of grain orientation in Section II

(B) Decreasing section thickness

(C) Section thickness measured at center of tensile specimen gage length

TABLE IV

FRACTURE TOUGHNESS PROPERTIES OF HP 9-4-25 STEEL  
ALLOY SOLID ROCKET MOTOR CASE

Temperature (°F)	Section Thickness (inches)	Direction (A)	$K_{IC}$ (KSI $\sqrt{\text{in.}}$ )
Room	1.4 1.0 1.0 } (B) 0.4 0.4	Axial (WR)	113.5 118.5 126.0 72.8(D) 73.9(D)
-65	1.4 1.4 } (B) 0.4	Axial (WR)	125.6 111.5 61.2(D)
Room	1.4 1.4 1.4 } (C) 1.0 0.4 0.4	Hoop (RW)	117.3 113.9 108.7 121.2 57.0(D) 53.1(D)
-65	1.4 1.0 } (C) 0.4 0.4	Hoop (RW)	111.7 99.0 74.4(D) 71.6(D)
Room	1.5 1.5	Short Hoop (RT)	88.9(D) 85.8(D)
-65	1.5 1.5	Short Hoop (RT)	87.7(D) 91.3(D)
Room	1.5 } (C) 1.5	Short Axial (RT)	93.9 94.4
-65	1.5 (C)	Short Axial (WT)	83.4(D)

(A) See explanation of grain orientation in Section II

(B) Section thickness measured at plane through which specimen fractured

(C) Section thickness measured at specimen notch tip

(D) The ASTM criterion  $B \geq 2.5 (K_{IC}/\sigma_{ys})^2$  was violated

The fatigue crack growth rate versus the stress intensity factor range for the rectangular DCB and the Mostovy DCB are presented in Figure 11 and Figure 12, respectively. Fatigue crack growth rate data was essentially the same for both types of specimen and was also compatible with other high strength steel data presented in the literature from a  $\Delta K$  value of 29.0 to a  $\Delta K$  value of 50.0. The fracture surfaces of representative rectangular DCB and Mostovy DCB specimens tested in laboratory air are shown in Figures 13 and 14, respectively. The fatigued fracture surfaces were characteristically flat.

Neither a distilled water environment nor variations in the relative humidity of the laboratory air appreciably influenced the fatigue crack growth behavior (see Figure 12). Accelerated environmental crack growth which occurs in some high strength steels was not observed in HP 9-4-25 steel. This could be due to one or more of three apparent factors: fatigue loading at 60 cpm does not allow the stress corrosion mechanism sufficient time to exert an influence on the crack growth rate; the fatigue mechanism is the overriding controlling factor of crack growth in this alloy; or a slightly humid environment is as detrimental as a water environment. The fracture surface of the fatigue crack growth specimen tested in distilled water is shown in Figure 15. The fatigued fracture surface is generally flat.

Statically loaded SCC properties are presented in Figure 16. A comparison of this data with data generated by Harmsworth and Cervay<sup>3</sup> (see Figure 17) using medium toughness D6ac steel shows the HP 9-4-25 steel to be significantly less sensitive to an aqueous environment under static load conditions.

A quite discernable difference was observed in the behavior of the  $K_I$ -dependent-of-crack-length (rectangular) specimens and the  $K_I$ -independent-of-crack-length (Mostovy) specimens under SCC conditions. Both types of specimen experienced an incubation period and slow subcritical crack growth. However, while the Mostovy specimen exhibited an unexpected crack arrest, the crack growth in the rectangular specimen continued on normally until fracture. The slow subcritical crack growth rate data obtained from the Mostovy specimen prior to arrest was very erratic (see Figure 16). Fracture surfaces of statically loaded rectangular DCB and Mostovy DCB specimens tested in distilled water are shown in Figures 18 and 19, respectively. The fracture surfaces are generally flat.

A corroded precrack was shown to only affect the incubation period and not the crack growth rates (see Figure 16).

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<sup>3</sup>Harmsworth, C. L., and Cervay, R. R., "Fracture Toughness Evaluation of D6ac Steel in Support of the F-111 Aircraft Recovery Program," Tech. Memo AFML/LAE-71-2.

The HP 9-4-25 steel alloy is attractive for use in structural members for the following reasons. First, the plane strain flaw sensitivity index (FSI),

$$\left( \frac{K_{IC}}{\sigma_{ys}} \right)^2$$

of this alloy is 0.510 inches in the axial direction (crack parallel to longitudinal grain direction). This FSI is for the low tensile strength-high toughness unworked material. This compares favorably with other high strength steel alloys having the same yield strength (see Figure 20). Since the material's critical flaw size is proportional to the FSI, it is of prime concern from a structural integrity viewpoint to keep the index as high as possible. As expected, the highly worked roll-extruded material appears to have a lower FSI value than the unworked material. But, due to material thickness limitations, the plane strain FSI was not evaluated for both states of the material. The second advantage of the HP 9-4-25 is that the fatigue crack growth behavior of this alloy is relatively stable and generally superior to other steels of comparable strength in an aqueous environment. In air the fatigue crack growth properties are comparable to other steels up to a  $\Delta K$  value of 50.0. Static load SCC crack growth in an aqueous environment is also equal or superior to comparable steels.

## SECTION V

### SUMMARY

The thin section highly worked HP 9-4-25 steel alloy displayed an increase in tensile strength and an apparent decrease in fracture toughness when compared with the thick section unprocessed material of the experimental SRM case. The exact reduction in toughness due to processing could not be definitely substantiated because of thickness limitations in the processed section. That is, the thickness of the material was not sufficient by ASTM standards for a valid  $K_{IC}$  test. However, the toughness values calculated are sufficiently lower in the thin section than in the thick section to say that the thin section has lower fracture toughness. It is not conclusive that the degradation of toughness negates the other desirable attributes of the process.

Fatigue crack growth properties of the processed HP 9-4-25 at the lower  $\Delta K$  levels in both air and distilled water environment were comparable to other high strength steels. Acceleration of growth rate was not discernable in an aqueous solution under fatigue loading. Statically loaded SCC properties were excellent when compared to medium toughness D6ac steel alloy.

Because of its relative insensitivity to crack acceleration in an aqueous environment and its excellent flaw sensitivity index, the HP 9-4-25 steel alloy is attractive for use in structural members.

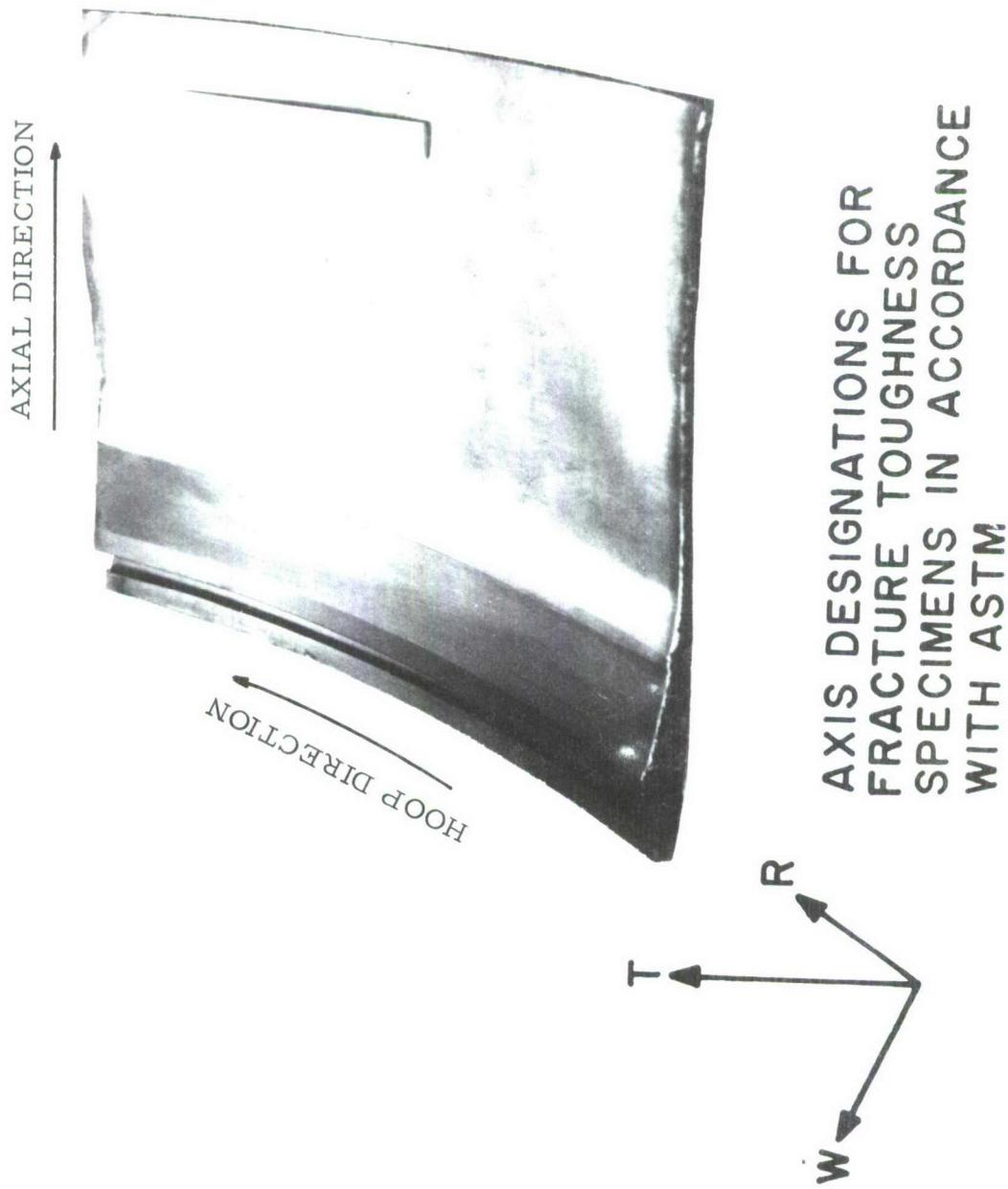


Figure 1. HP 9-4-25 Test Section from Roll-Extrusion Processed Ring S/N 004

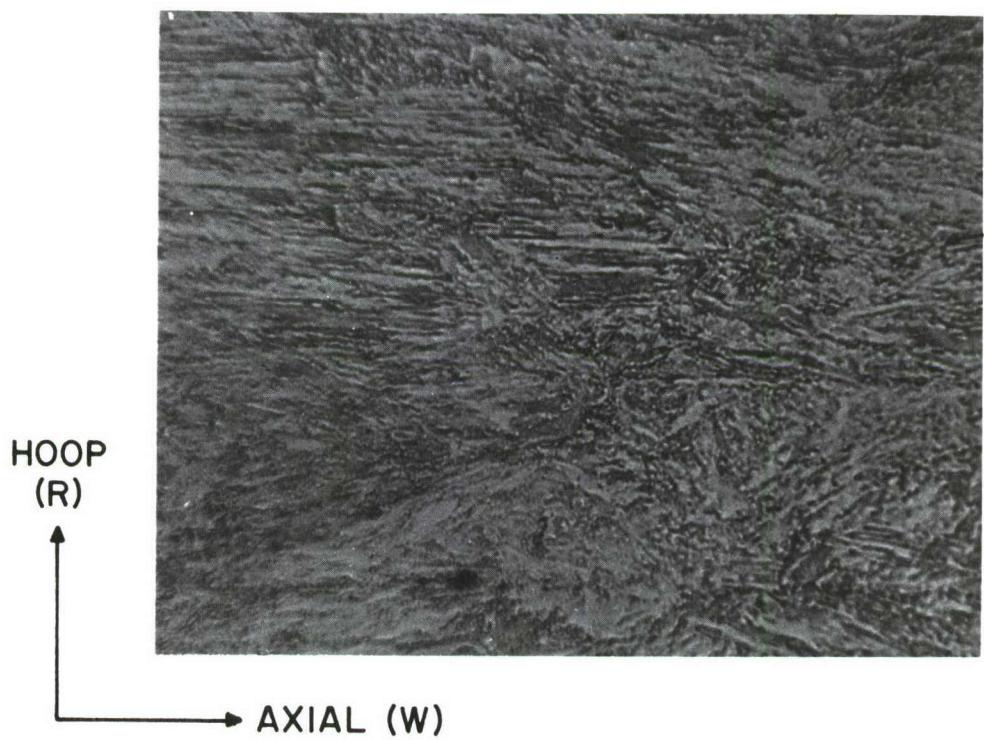


Figure 2. Photomicrograph at 750X of the Highly Worked Region of the SRM Case

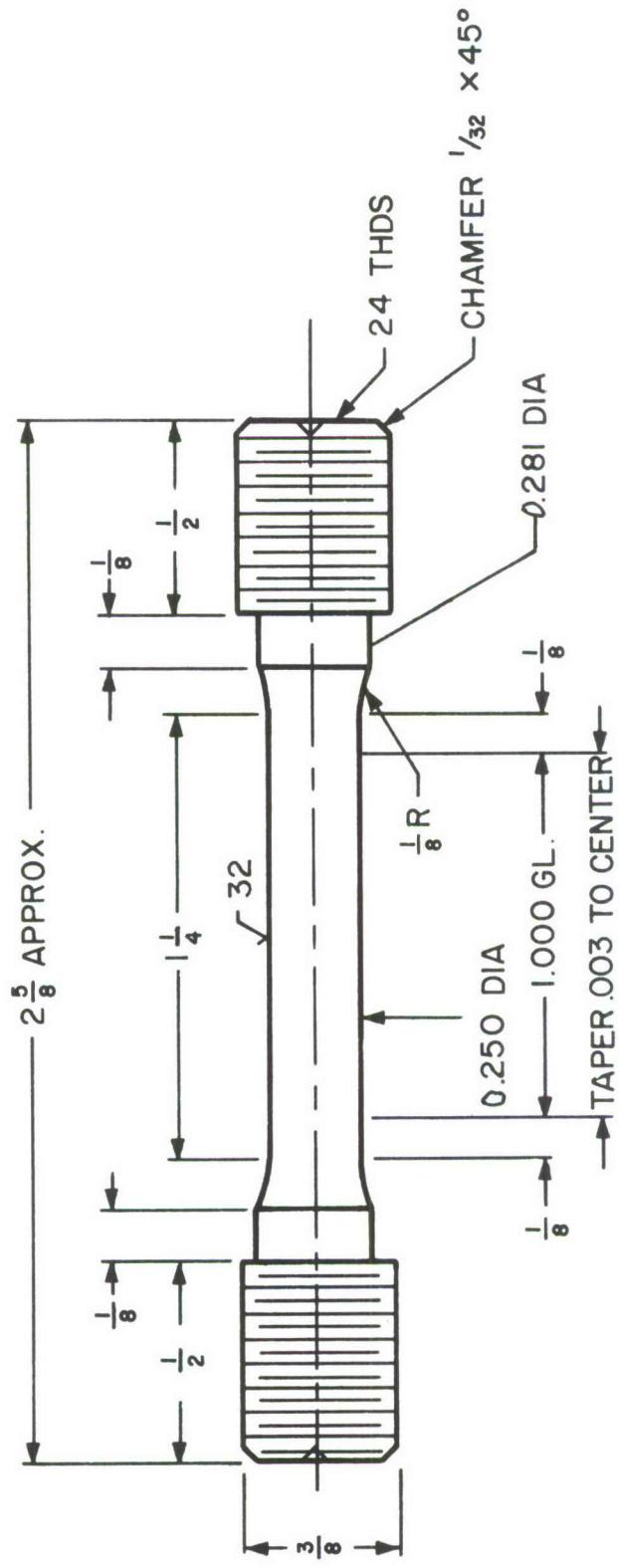
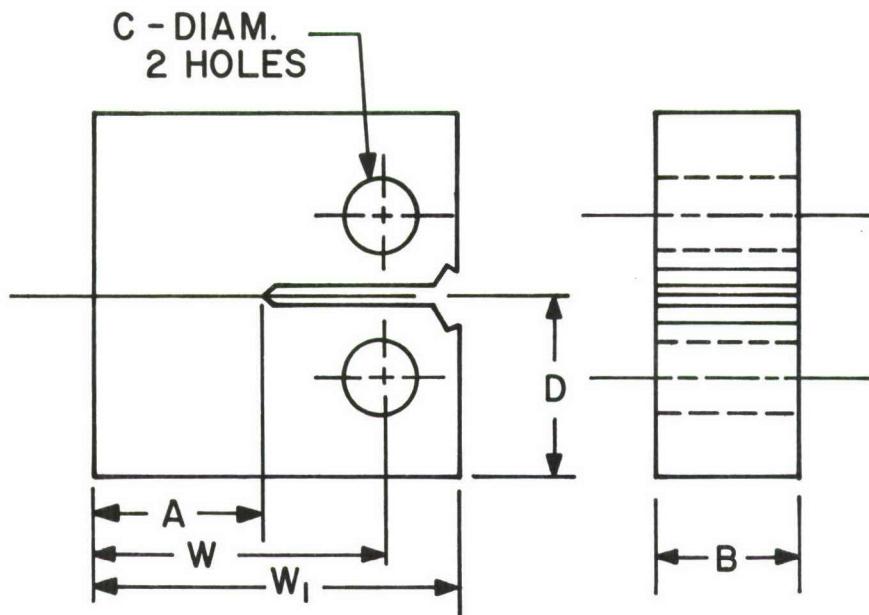


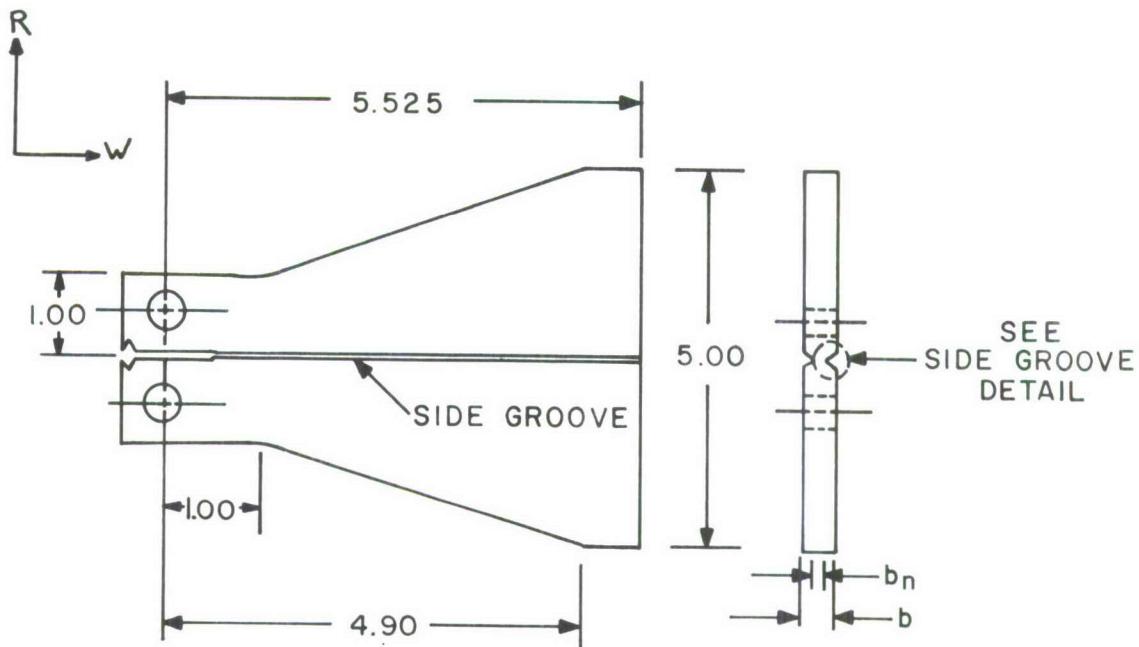
Figure 3. Tensile Specimen Configuration



#### DIMENSIONS

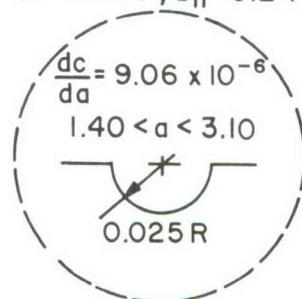
SPECIMEN THICKNESS (INCHES)	A	B	W	W <sub>1</sub>	D	C
1	1.100	1.000	2.000	2.500	1.200	0.500
3/4	0.835	0.750	1.500	1.875	0.900	0.375
1/2	0.550	0.500	1.000	1.250	0.600	0.250

Figure 4. Compact Tension Fracture Toughness Specimen Configuration

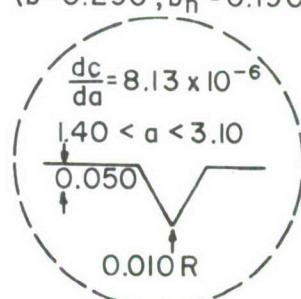


### SIDE GROOVE DETAIL

FATIGUE CRACK GROWTH  
( $b = 0.290$ ,  $b_n = 0.240$ )



CORROSION CRACK GROWTH  
( $b = 0.290$ ,  $b_n = 0.190$ )



$$K_I = P \sqrt{\frac{E(dc/da)}{2b_n(1-\mu^2)}}$$

WHERE:     $K_I$  = STRESS INTENSITY FACTOR (ksi  $\sqrt{\text{in}}$ )  
 P = LOAD (kips)  
 E = YOUNG'S MODULUS (ksi)  
 c = COMPLIANCE (kips/in)  
 a = CRACK LENGTH (in)  
 b = GROSS THICKNESS (in)  
 $b_n$  = NET THICKNESS (in)  
 $\mu$  = POISSON'S RATIO

Figure 5. Mostovy Tapered DCB Crack Growth Specimen Configuration ( $K_I$  Independent of Crack Length)

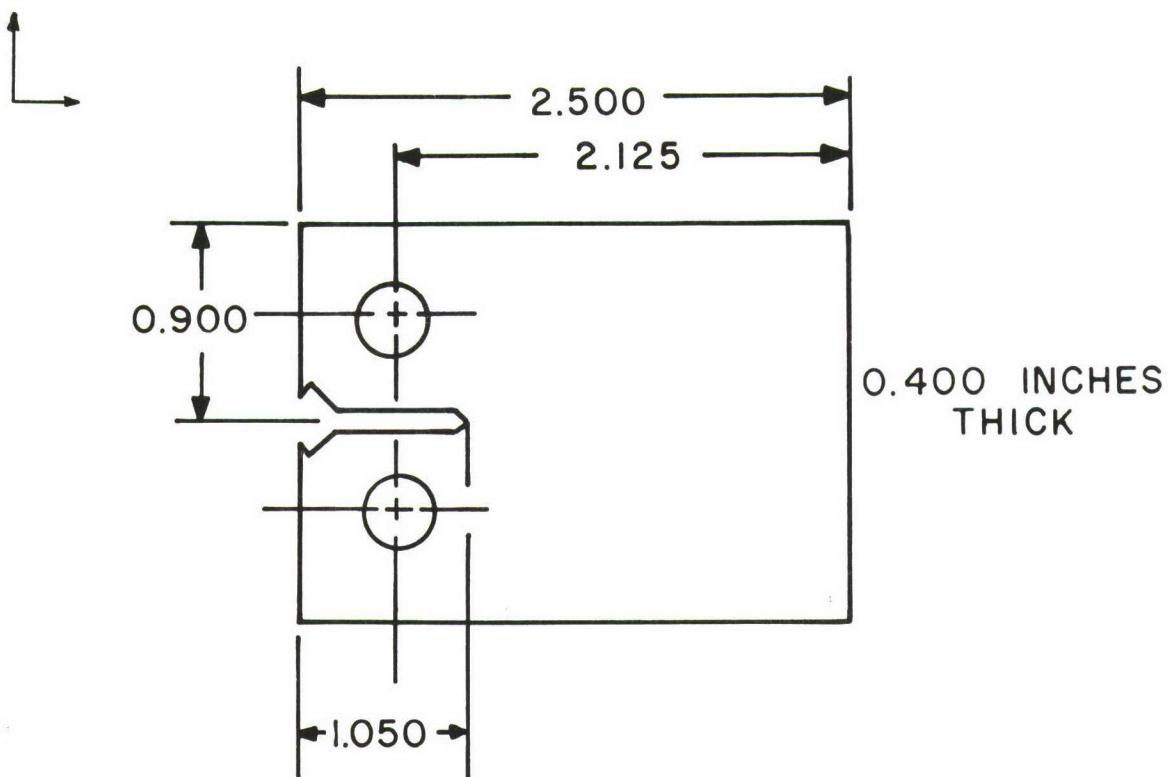


Figure 6. Rectangular DCB Crack Growth Specimen Configuration ( $K_I$  Dependent upon Crack Length)

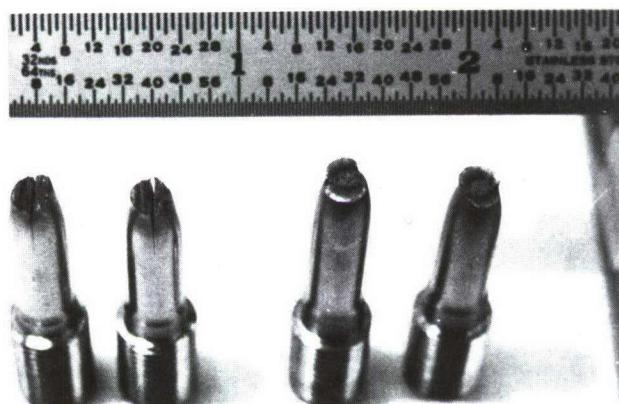
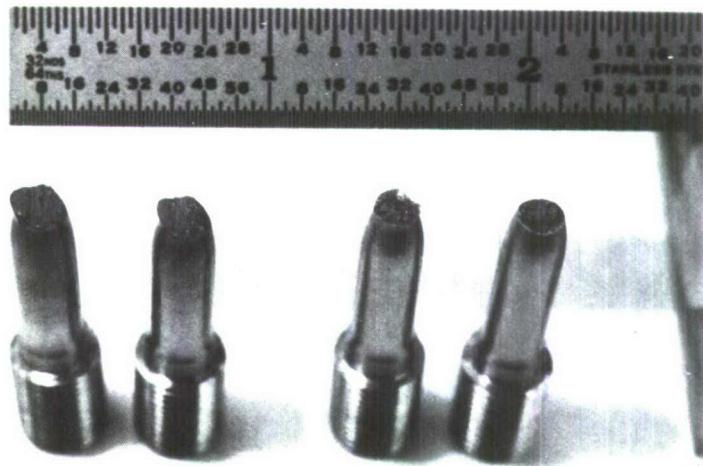


Figure 7. Fractured Tensile Specimens. Right: Axial direction, room temperature test, removed from 1.4 inch thick section. Left: Axial direction, room temperature test, removed from 0.4 inch thick section.



P17 ↴  
Figure 8. Fractured Tensile Specimens. Right: Hoop direction, room temperature test, removed from 1.4-inch-thick section. Left: Hoop direction, room temperature test, removed from 0.4-inch-thick section.

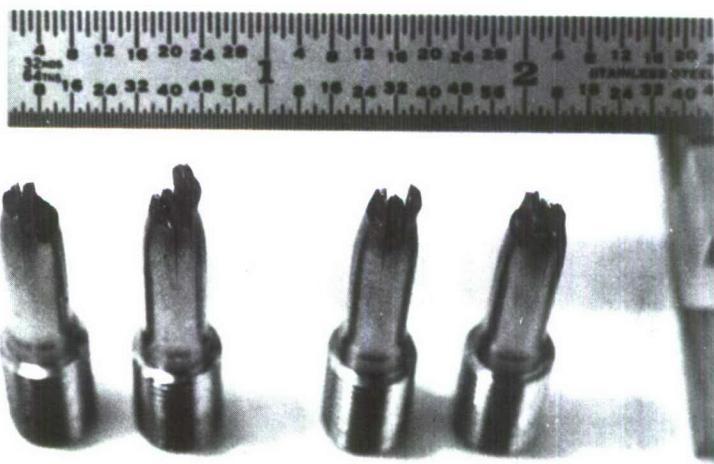


Figure 9. Fractured Tensile Specimens. Right: Axial direction, -65°F test temperature, removed from 0.4-inch-thick section. Left: Axial direction, -65°F test temperature, removed from 0.4-inch-thick section.

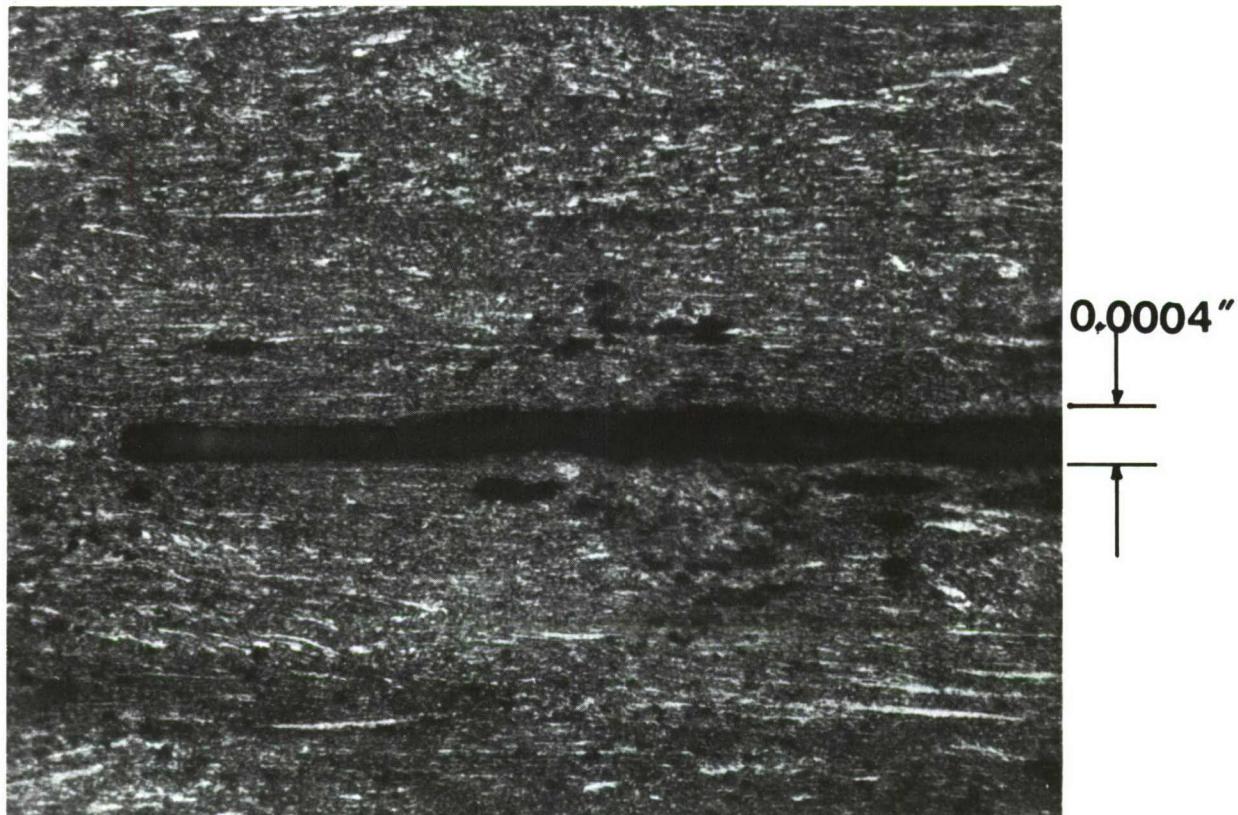


Figure 10. Photomicrograph of Laminations in Axial Tensile Specimen (600X).

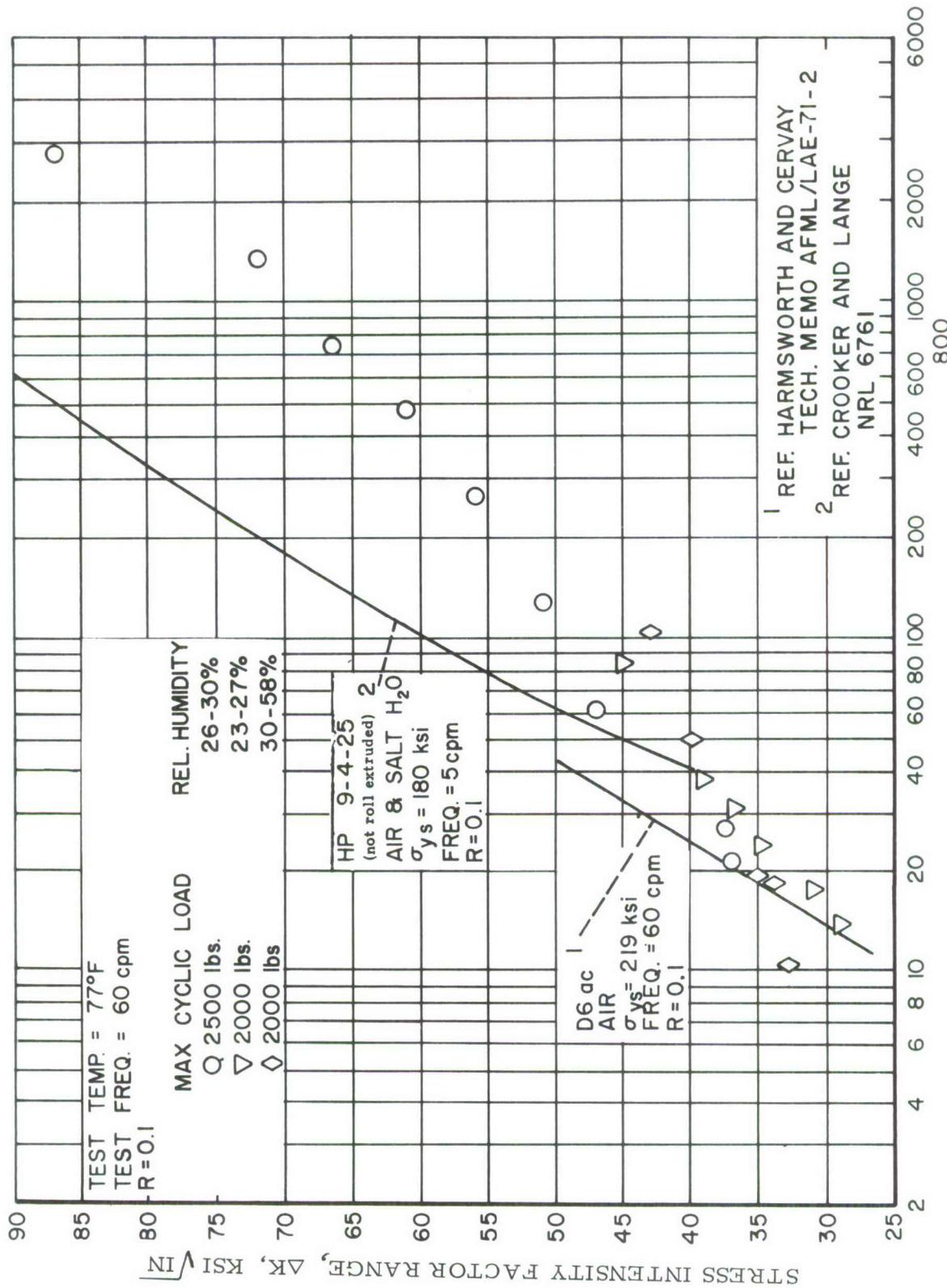
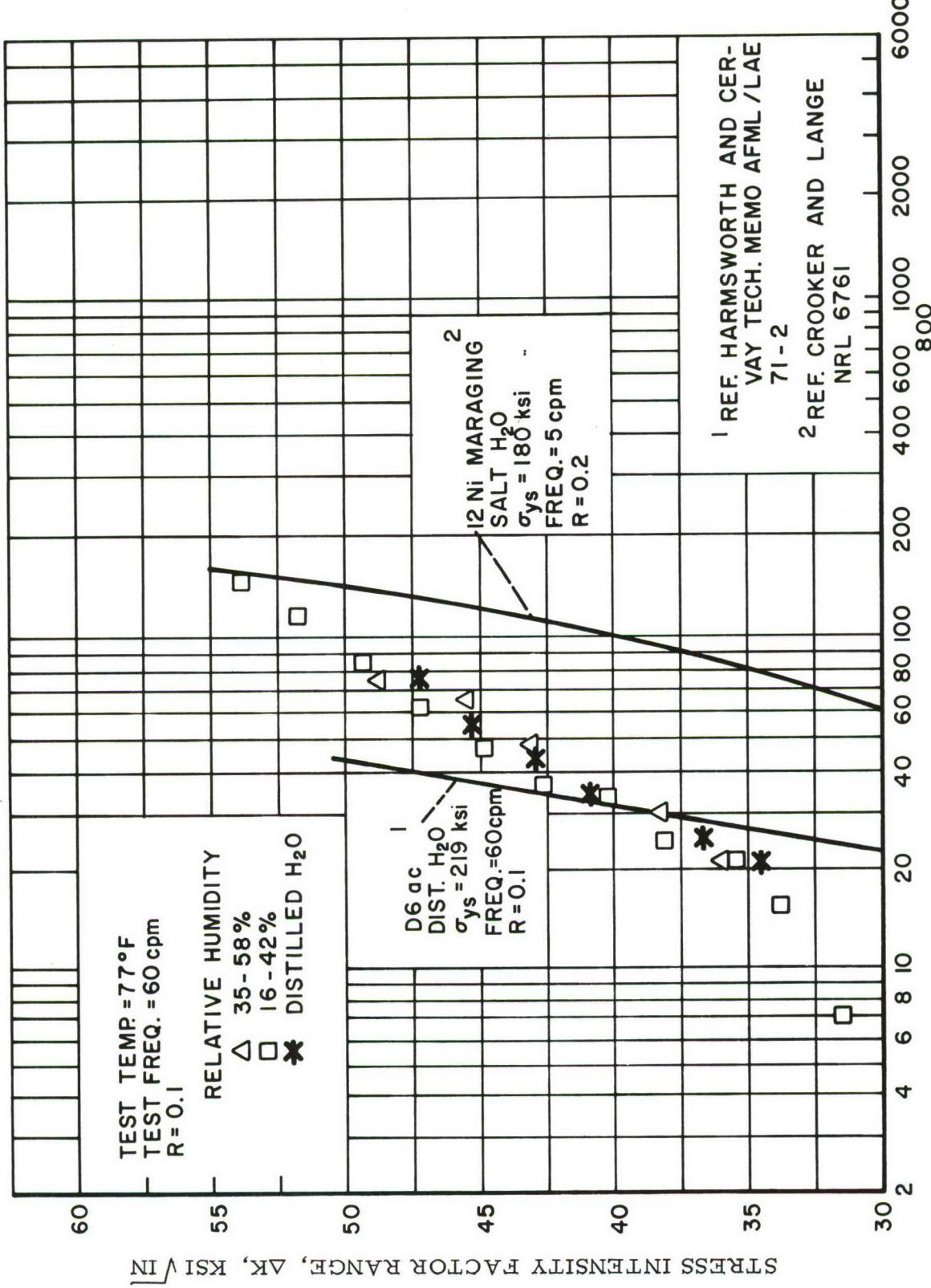


Figure 11. Stress Intensity Factor Range Versus Fatigue Crack Growth Rate for Roll Extruded HP 9-4-25 Steel, Rectangular DCB Specimen, with Other Data



## Stress Intensity Factor Range Versus Fatigue Crack Growth Rate for HP 9-4-25 Steel, Mostovoy DCB Specimen, with Other Data

Figure 12.

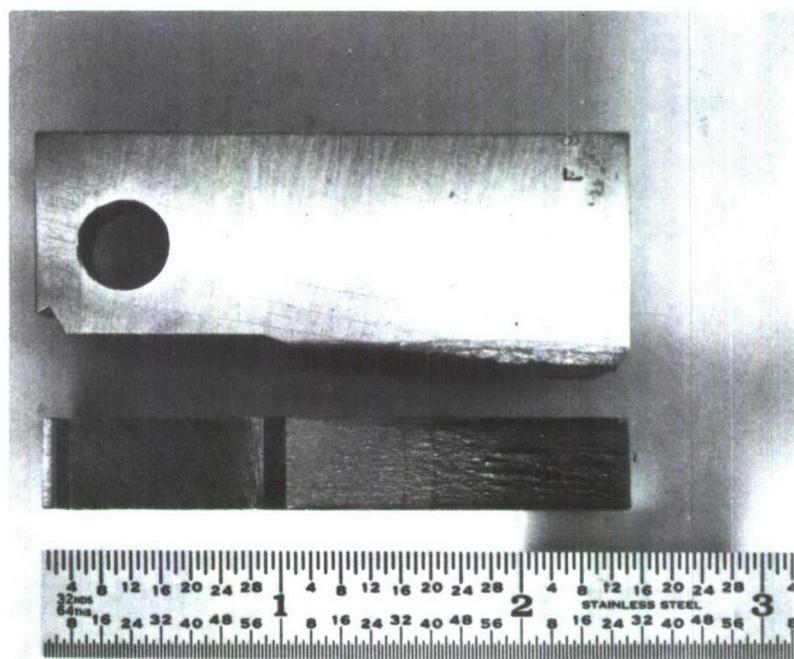


Figure 13. Fracture Surface of a Fatigue Crack Growth Rectangular DCB Specimen Tested in Laboratory Environment

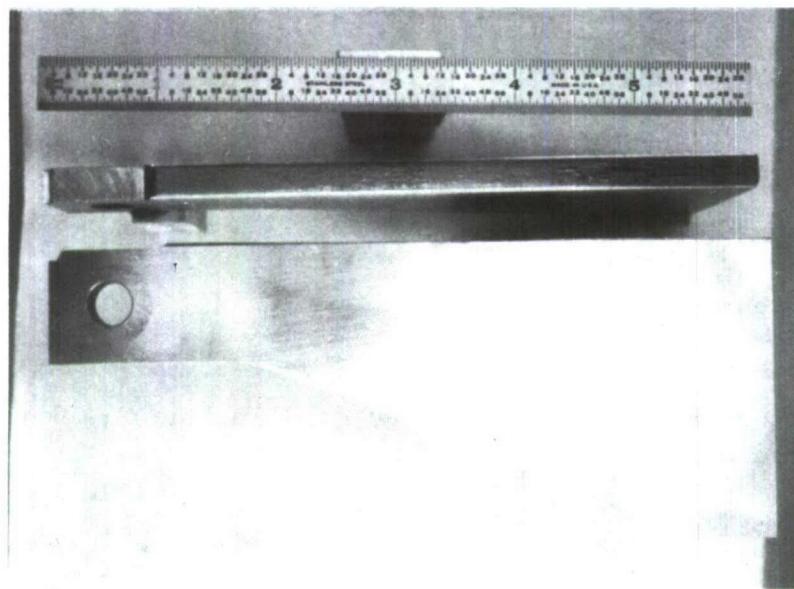


Figure 14. Fracture Surface of a Fatigue Crack Growth Mostovoy DCB Specimen Tested in Laboratory Environment

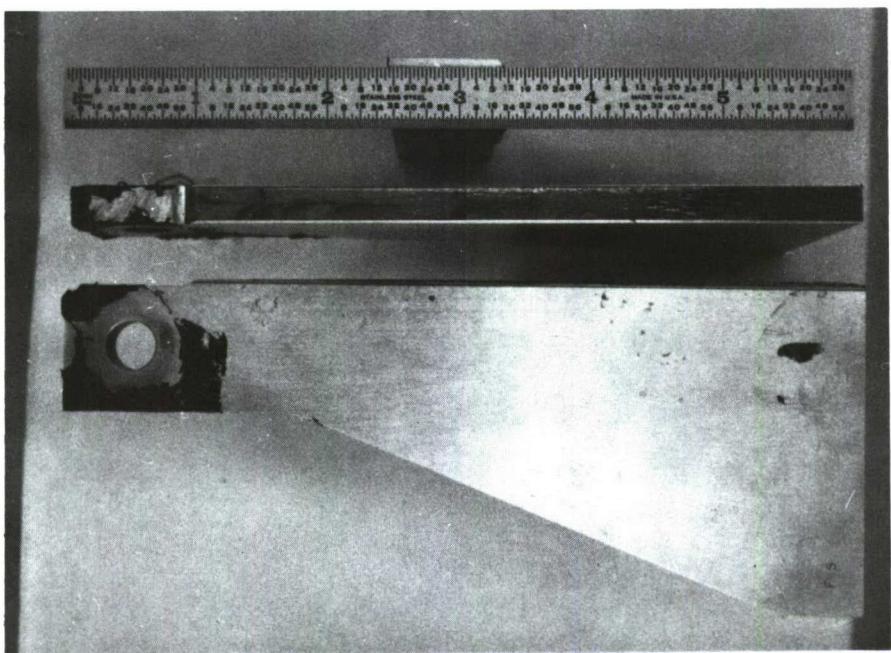


Figure 15. Fracture Surface of a Mostovoy DCB Fatigue Crack Growth Specimen Tested in Distilled Water

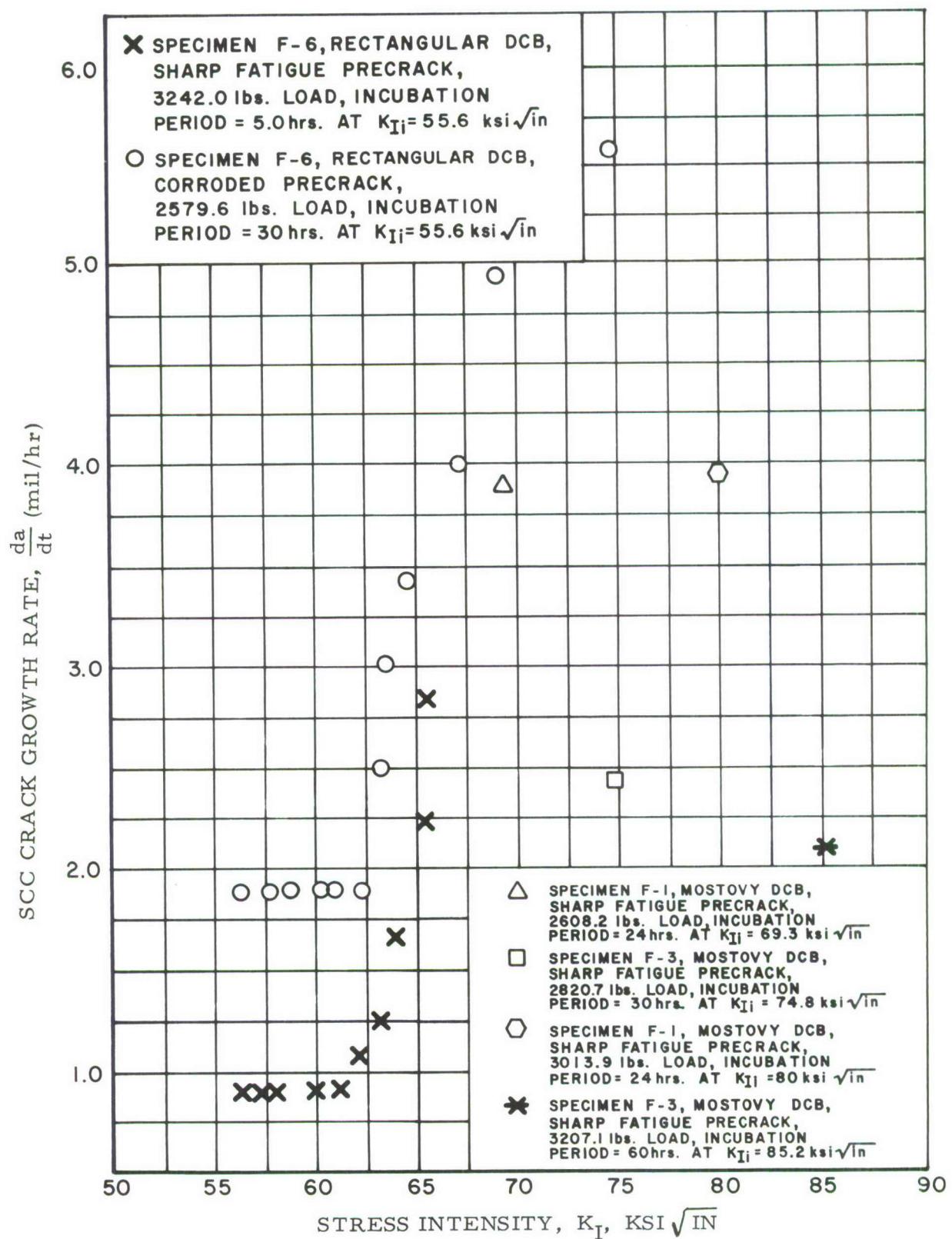


Figure 16. SCC Crack Growth Rate Versus Stress Intensity for HP 9-4-25 Steel

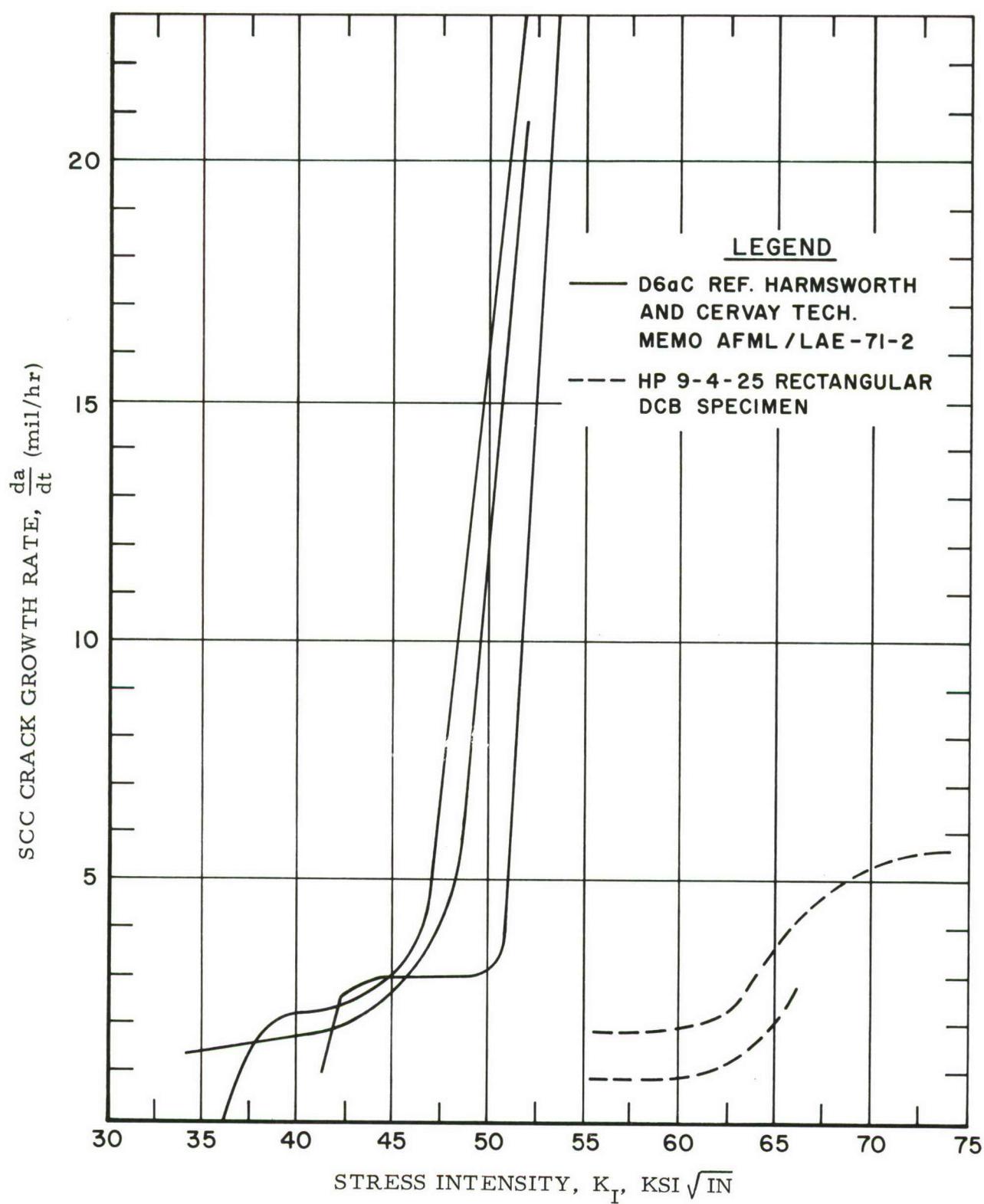


Figure 17. SCC Crack Growth Rate Versus Stress Intensity for D6ac and HP 9-4-25

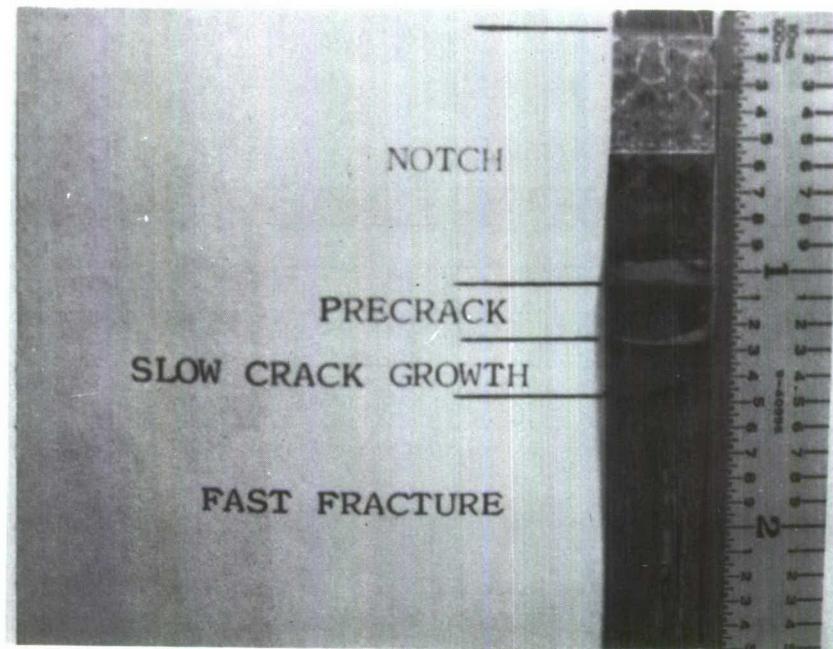


Figure 18. Fracture Surface of a Statically Loaded Rectangular DCB Crack Growth Specimen Tested in Distilled Water

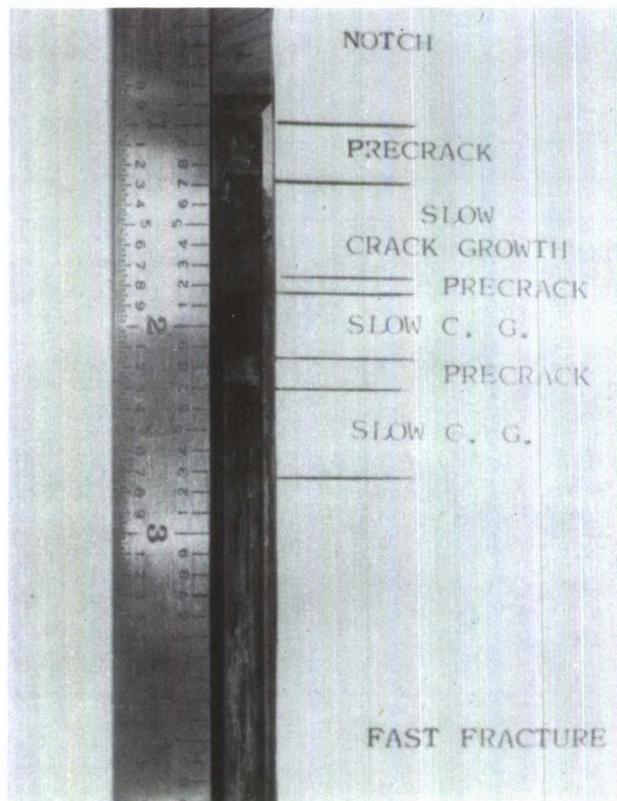


Figure 19. Fracture Surface of a Statically Loaded Mostovoy DCB Crack Growth Specimen Tested in Distilled Water

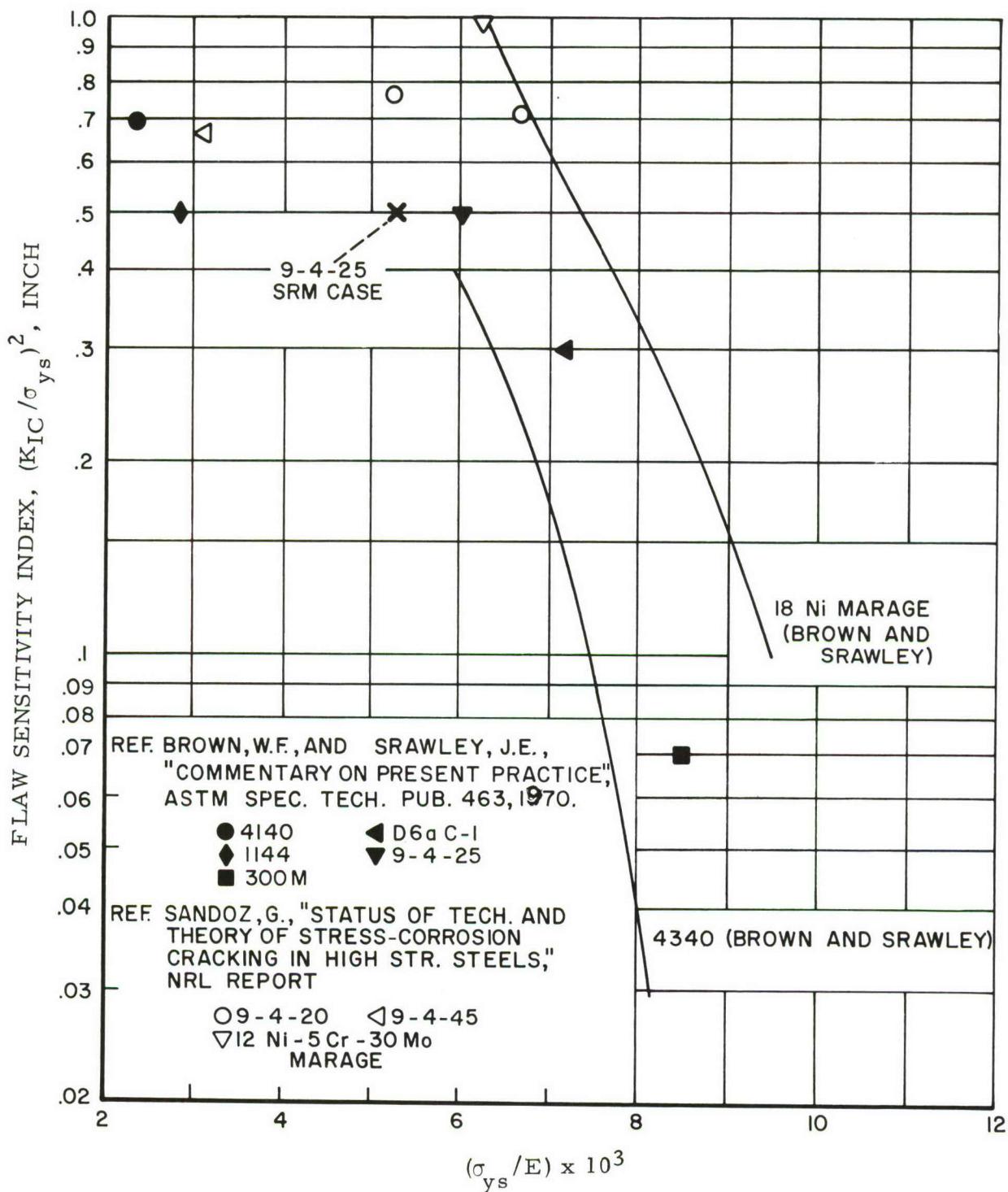


Figure 20. Flaw Sensitivity Index Versus Yield Strength Normalized by Young's Modulus for Several Steel Alloys

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13. ABSTRACT  A section of internal roll-extruded NP 9 Ni-4Co-25C steel alloy was obtained from an experimental 120-inch rocket motor case for Titan III and sent to the AFML for evaluation by the Space And Missile System Organization (SAMSO). Tensile and fracture toughness properties were obtained at room temperature and at -65°F. Crack growth properties were obtained at room temperature in laboratory air and in distilled water. An increase in tensile strength and a decrease in ductility were observed to occur with increased cold forming reduction. Fracture toughness properties appeared to decrease with increased rolling reduction. Fatigue crack growth properties in laboratory air were compatible with other high strength steels for the lower $\Delta K$ values. Fatigue crack growth properties in distilled water showed no acceleration due to aqueous environment influences. Statically loaded stress corrosion crack (SCC) growth properties were an improvement over available D6ac steel alloy data.		

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